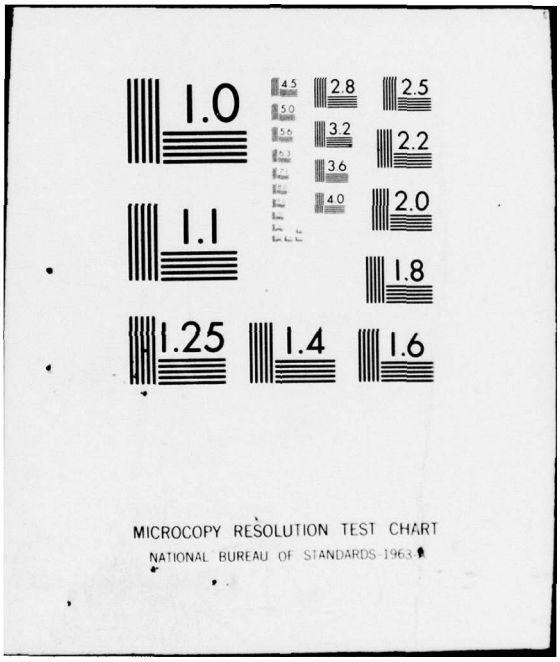
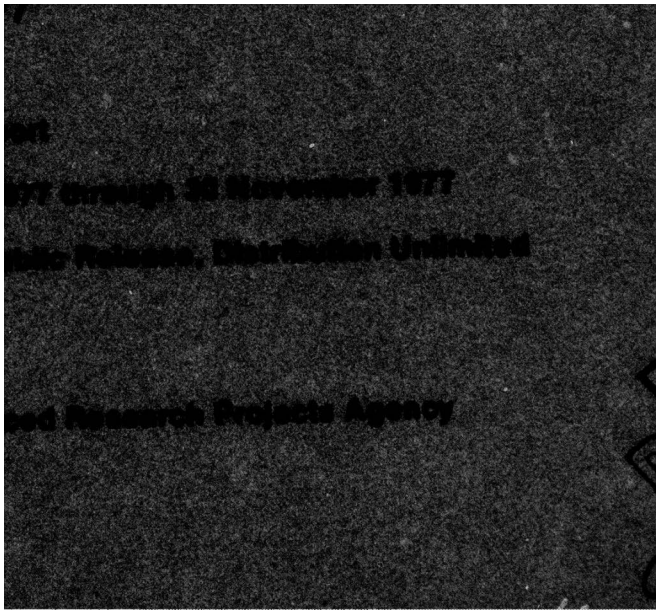
AD-A049 469 PRATT AND WHITNEY AIRCRAFT GROUP WEST PALM BEACH FLA --ETC F/G 13/8
APPLICATION OF SUPERPLASTIC STEELS.(U)
DEC 77 E R SLAUGHTER, R G BOURDEAU F33615-77-C-5114
UNCLASSIFIED FR-9489 NL

1 OF 1
AD A049469

END
DATE FILMED
3-78
DDC


1.0
4.5
5.0
5.6
6.3
2.8
3.2
3.6
4.0
2.5
2.2
2.0
1.8
1.1
1.25
1.4
1.6
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A



UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
(6) APPLICATION OF SUPERPLASTIC STEELS.

5. TYPE OF REPORT & PERIOD COVERED
(9) Semiannual Progress Report.
1 Jun[redacted]-30 Nov[redacted]77

6. PERFORMING ORG. REPORT NUMBER
(14) FR-9489

7. AUTHOR(s)
(10) E. R. Slaughter
R. G. Bourdeau

8. CONTRACT OR GRANT NUMBER(s)
(15) F33615-77-C-5114

9. PERFORMING ORGANIZATION NAME AND ADDRESS
United Technologies Corporation
Pratt & Whitney Aircraft Group
Government Products Division
Box 2691, West Palm Beach, Florida 33402

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS

11. CONTROLLING OFFICE NAME AND ADDRESS
Defense Advanced Research Projects Agency
1400 Wilson Boulevard
Arlington, Virginia 22209
(Dr. E. C. vanReuth)

12. REPORT DATE
(11) Dec[redacted]77

13. NUMBER OF PAGES
(12) 18 p.
16

14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*
Air Force Materials Laboratories
Wright-Patterson AFB, Ohio 45433
(Mr. A. Adair)

15. SECURITY CLASS. *(of this report)*

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*
Approved for Public Release, Distribution Unlimited

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*
Superplastic Steels, Forging, Powder Metallurgy, Rapid Solidification, Ultrahigh Carbon, GATORIZING™

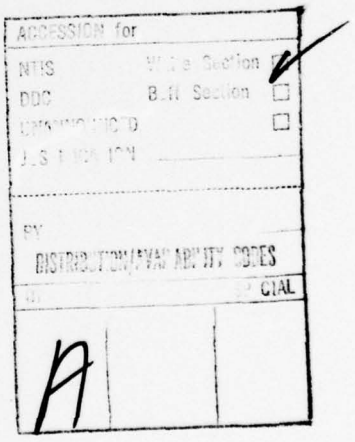
20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*
This program is being conducted to demonstrate that the GATORIZING™ forge process can be applied to the precision forging of superplastic steels, and to determine the economic benefits gained over conventional forging and subsequent machining to final shape. During this report period, the thermomechanical processing of an ultrahigh-carbon steel was initiated to convert its cast structure to a characteristic fine grained superplastic structure. Alloy development studies were initiated with laser treated arc cast buttons which were extruded and thermomechanically processed in such a way that the stability of recrystallized fine grained structures could be evaluated.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE
S/N 0102-LF-014-6601

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

392 887

CONTENTS



ILLUSTRATIONS

SUMMARY

The thermomechanical processing of an ultrahigh-carbon (1.6% C) steel to convert its cast structure to a fine grained structure was performed on commercial equipment and on a scale which demonstrates the feasibility of the process. The forged material obtained is presently being evaluated with respect to structure, strain rate sensitivity, total elongation and forgeability.

A necessary condition of superplasticity, a very fine grain size, was attained in boron containing ferrous alloys by rapid solidification plus thermomechanical processing. These fine grained structures were produced by a convenient experimental technique which roughly simulates, on samples weighing only milligrams, the thermomechanical processing that might be employed to produce large superplastic forgings. The superplastic properties of many alloys degrade during superplastic deformation because of grain growth; an experimental alloy appeared to be superior to a control alloy in resisting such growth.

SECTION I

INTRODUCTION

Superplasticity in metal alloys permits forming to complex shapes and at relatively low stress levels. In some cases, as with nickel-base superalloys, superplasticity makes it possible to forge to complex shapes a material which is considered unforgeable. In other cases, as with a Zn-Al alloy, forming operations used for plastics are employed to produce intricate parts to close tolerances and with faithfully reproduced surface textures. For steels, made superplastic, economics from processing and subsequent machining should be attainable and it can be expected that gains in properties associated with a very fine grain size may also be in the offing.

Research conducted at Stanford University by O. Sherby et al[1] has shown that superplasticity can be achieved in simple but ultrahigh-carbon steels (1.3 to 1.9 weight% C) by thermomechanical processing in such a way that a very fine grain size in the micron range is produced. The very high carbon contents are required to produce sufficiently high concentrations of cementite (20 to 29 volume % Fe_3C) to stabilize the ferrite at working and superplastic deformation temperatures. By working in the austenite-cementite range, the massive cementite phase present in the original casting is broken up and spheroidized. And by further working in the ferrite-cementite range, the transformed austenite or pearlite is also spheroidized, while the structure is refined to a very small grain size. Various thermomechanical methods were employed to produce grain refinement but it all cases the final structure, for superplastic behavior, was an equiaxed ferrite grain in the order of 1 to 3 microns stabilized with a submicron spheroidized cementite precipitate.

A material can be defined as superplastic if the strain rate sensitivity exponent, m, in the relation $\sigma = K \dot{\epsilon}^{m}$ (σ is the flow stress, $\dot{\epsilon}$ is the strain rate and K is a material constant) approaches 0.5 and high elongations (to 500%) are achieved. According to Stanford University researchers, the ultrahigh-carbon steels are superplastic at warm temperatures (below the γ to α + Fe_3C transformation temperature) at a strain rate in the order of 1% per minute.

The purpose of this Advanced Research Project Agency (ARPA) sponsored program is to apply the principles developed at Stanford University to the precision forging of steel shapes by the GATORIZING™ forging process, and to determine the benefits to be realized by the use of superplasticity in steels. Three parts, which are believed to be representative of mass produced items, were selected to evaluate the forging of superplastic steels. These are: (1) an aft closure of a laser guided missile, (2) a forward control section of the same missile, and (3) a die assembly for cold forming pinion gears.

The program is a 27-month effort starting with an evaluation of the forge-ability of an ultra high carbon steel composition produced initially as a cast billet. Alternately, the same alloy is to be produced as a powder to determine the advantages gained by the elimination of massive cementite. In conjunction with the above tasks is an evaluation of the potential of alloying to alter mechanical properties without loss of superplasticity. Finally, subsequent to a precision forge evaluation and component fabrication, the economic and material benefits derived from superplasticity in steel will be assessed.

This report is the first semiannual technical report, covering the period from 1 June 1977 through 30 November 1977. It deals with the preparation of a steel composition cast in billet form and with thermomechanical processing to convert it to a fine grained superplastic state. It also deals with the alloy development phase of the program.

[1] Sherby, Oleg D. et al, "A Summary Report on Superplastic Ultrahigh-Carbon Steels," Advanced Research Project Agency, Grant No. DAHC-15-73-G15, February 1977

SECTION II

PROCESS AND ALLOY DEVELOPMENT

PROCESS DEVELOPMENT

This phase of the program deals with the thermomechanical processing of cast steel to convert its structure to a superplastic fine grain structure as characterized by O. Sherby of Stanford University. Both the composition of the ultrahigh-carbon steel employed and the breakdown or thermomechanical processing parameters employed were as recommended by Prof. Sherby and his colleagues. The superplastic ultrahigh-carbon steel which they developed was processed almost entirely by the flat rolling of small slabs under laboratory conditions. Our initial attempt at scale-up was performed on 6-inch diameter ingots forged in a commercial 8000 lb steam hammer forge at the Ladish Corporation.

The nominal composition of the ultrahigh-carbon steel which was vacuum induction melted and cast in our facilities is as follows:

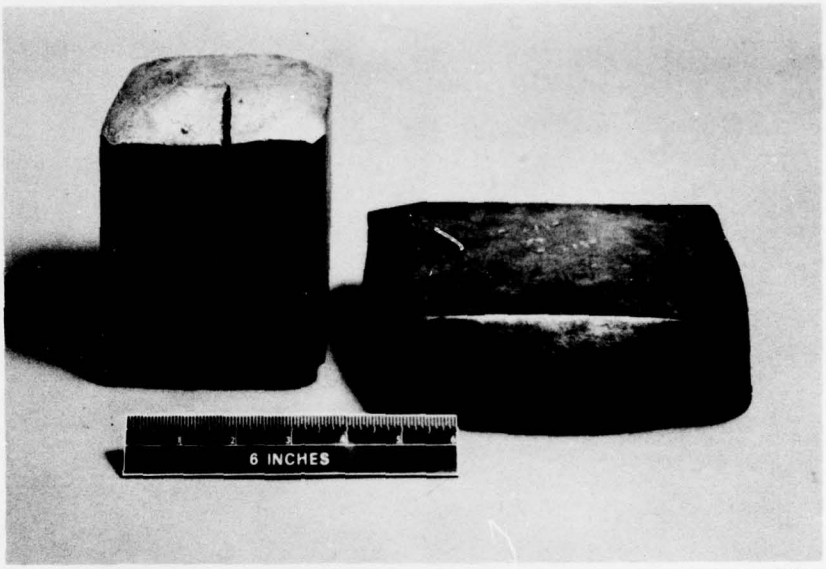
carbon	1.5%
manganese	1.0%
vanadium	0.15%
silicon	0.1% maximum
iron	balance

Eight 38 kg heats of this nominal composition were produced and the ingots were lathe turned to condition their surfaces for forging. The ingots were to be hammer forged by the idealized process involving two forging stages; the ingots were to be forged to a true strain of 1.0 at 1560°F (850°C) in the first stage and forged to a strain of 1.4 at 1200°F (650°C) in the second stage. The temperature of the billets was to be lowered sufficiently between forging stages to allow transformation of the austenite.

For the first stage, the ingots were heated in a 1560°F (850°C) furnace for 2 hours, then drawn, upset and cubed without reheating. The starting temperature of the ingots for forging as measured by a contact pyrometer was approximately 1480°F (80°C); the finish temperature was 1410°F to 1500°F (765° to 850°C) i.e., the temperature increased during forging for some ingots but decreased for others. After the first stage of forging, the billets were air cooled to room temperature and conditioned to remove small cracks.

The second forging stage involved a drawing and an upsetting operation with four reheating operations. The billets were preheated for approximately 2 hours in a 1200°F (650°C) furnace. At the start of the draw operation, the billet temperature was 1100°F (593°C). The draw was completed and the upsetting begun before the first reheat. At that time the billet temperatures were 1000° to 1050°F (538° to 565°C). The billets were reheated to approximately 1100°F (593°C) in 15 to 30 minutes and the upsetting continued; this cycle was repeated three times. The finish temperatures were 950° to 1000°F (510° to 538°C). Four of the billets ruptured during upsetting and were dropped from the process. Four were completed with only minor cracks visible, as shown in Figure 1.

During the second forging stage, each 30 lb (13.5 kg) billet was struck approximately 240 blows (60 per reheat) with approximately 40% of the maximum energy of the 8000 lb steam hammer. Since a large amount of kinetic energy was supplied by the massive hammer to the small billets while their temperature was falling, it was apparent that only a small fraction of the energy of the hammer resulted in deformation of the billets.



FE 164074

Figure 1. Forged Ultrahigh-Carbon Steel Billets

The foregoing procedure was a first attempt to forge ultrahigh-carbon steel on commercial equipment and on a scale which demonstrates the feasibility of the process. Although there was concern that the cast billets might fracture prematurely, the results obtained prove that this type of material can be worked even below the austenite-pearlite transition temperature. However, this is not to imply that the forging cycle or the extent of deformation were optimum or even satisfactory for the alloy selected. The forged material produced is presently being evaluated with respect to structure, strain rate sensitivity, total elongation and forgeability.

An alternate approach to produce a fine grain ultrahigh-carbon steel in bulk or close to desired net shape forms is by powder metallurgy. Since rapidly solidified powders have a fine grain size, far less thermomechanical processing should be required to produce a fine grain superplastic structure. This approach will be initiated as soon as steel powders have been received from a producer of gas atomized metal and alloy powders.

ALLOY DEVELOPMENT

A very fine grain size is necessary for superplasticity. Generally, these very fine grains are achieved in the most abundant phase of the alloy by a fine dispersion of a second phase. Typically, the dispersion is created by a reaction in the solid state, for example, the well known zinc-aluminum superplastic alloy and ultrahigh-carbon steels fall in this category.

For the alloy development phase of this program, an approach is employed which is known to produce a fine grain size. Specifically, it is a method which employs a focussed laser beam to produce a molten surface layer on a test coupon which solidifies by conduction at rates in the order of those obtained during gas atomization of metal powders. This method is described more fully in Section III.

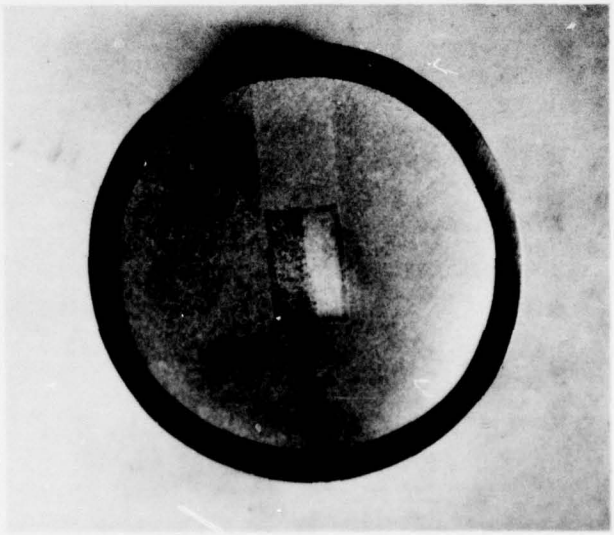
The evaluation of samples is limited because of the very small volume of laser treated material. However, the material can be thermomechanically treated and, as shown, laser passes can always be found and the material evaluated with respect to factors known to be important for superplastic behavior. The method appears to be most valuable for alloy development where many alloy compositions need to be studied.

Boron was judged the most promising alloying addition to be used in conjunction with rapid solidification; boron-rich phases are likely candidates for the dispersed phase. Boron is very soluble in liquid iron but virtually insoluble in ferrite and austenite.

More than a hundred pairs of button ingots comprising 83 compositions were melted. Most alloys contined 0.1% to 1.0% boron while the boron-free alloys were used as experimental controls. Most of these 55 gram button ingots are still being processed, however, three of the alloys have been processed by the complete sequence of operations listed below:

1. Nonconsumable arc melting of button ingots
2. Laser treating
3. Extruding
4. Annealing (optional)
5. Cold working
6. Annealing
7. Upsetting at a low strain rate.

The first three operations simulated the operations of gas atomization powder making and consolidation of the powder by extrusion. Button ingots were melted using elemental materials of moderate purity, for example, electrolytic iron and 99.8% boron. Pairs of laser treated button ingots were assembled for extrusion with the laser treated surfaces in contact to eliminate chemical potential gradients in the vicinity of the laser treated material during subsequent high-temperature operations. Since there were six pairs of buttons separated by tungsten markers in each extrusion can, it was necessary to choose common extrusion conditions for all alloys; the cans were preheated for 2 hours at 1600°F (872°C) and extruded at a nominal 7.8:1 extrusion ratio. A cross section of an extrusion is shown in Figure 2. The pair of button ingots was centered in the extrusion, surrounded by carbon steel and a stainless steel can.



Mag: 2X

FAL 45281A

Figure 2. Cross Section of an Extrusion

Two steels with 1.6% carbon, supplied through the courtesy of Prof. Sherby, were processed as experimental controls. It was intended that they be processed as nearly like one of the processes developed by Prof. Sherby and associates as possible within the framework of the processing sequence previously discussed. One of their processes included operations for spheroidizing a pearlitic structure by annealing, cold working and recrystallizing. The annealing operation at step 4 in the sequence of operation listing was intended to spheroidize the pearlite. The boron-containing alloys were not annealed at this point in the processing sequence.

The experimental alloys and controls were cold worked by upsetting 50%. This operation was followed by a recrystallization anneal. All alloys now processed were annealed for two hours at 1300°F (705°C), but it is intended that future annealing conditions be chosen generally to produce the minimum recrystallized grain size for each alloy. This grain size will be one of the major factors in selecting alloys for further study, including the following slow strain rate upsetting test. It is anticipated that the recrystallized grain size will be the minimum grain size for each alloy; therefore, alloys that do not have a sufficiently fine grain size at this stage will be abandoned.

The slow strain rate upsetting operation was intended to simulate a superplastic forming operation. Since many superplastic alloys suffer significant grain growth during low strain rate deformation in the superplastic temperature range with a resulting increase in flow stress, resistance to grain growth under these conditions is a desirable characteristic of a superplastic alloy. Two boron-containing alloys and a 1.6% carbon steel, which served as a control, were upset to a true strain of 2.15 (88.4% reduction in height) at a strain rate of 0.01/minute at 1290°F (700°C). The three specimens were upset simultaneously in a 100 ton capacity press with heated dies. After this operation, the rapidly solidified regions were easily identified metallographically from the remainder of the boron-containing alloys by an abrupt, drastic change in grain size and dispersed phase particle size, shape, and spacing at the interface, Figure 3.

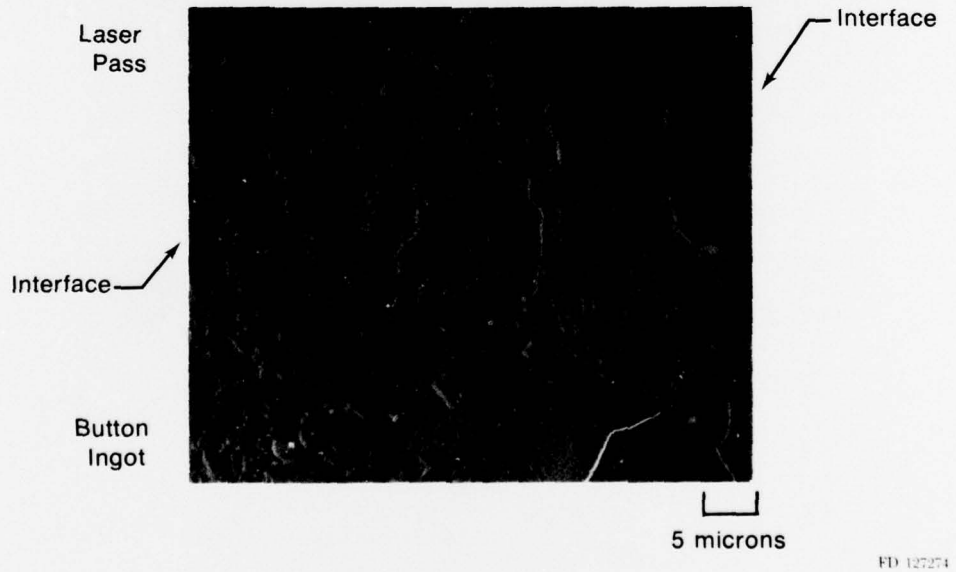


Figure 3. Interface Between Laser Pass and Button Ingot in Slow Strain Rate Test Specimen

Three alloys were processed completely: a binary iron, 1.0% boron alloy; a ternary iron 0.7% molybdenum, 1.0% boron alloy; and a 1.6% carbon steel. The binary alloy served as the prototype for boron-containing ferrous alloys. Preliminary company-sponsored experiments had indicated a potential of this alloy for the very fine grain sizes characteristic of superplastic alloys. As mentioned previously, this binary alloy is being procured as gas atomized powder to validate the use of the laser treatment in alloy development experiments for rapidly solidifed alloys. The ternary alloy was among the first modifications of the binary alloy, specifically the modification of the binary with a ferrite stabilizing addition. The carbon steel served primarily as an experimental control.

The solidification rate profoundly affected the structure of the extruded binary alloy. The rapidly solidified alloy had a finer ferrite grain size, smaller particles of a dispersed phase* and smaller interparticle spacing than the slowly solidified alloy. The dispersed phase of the slowly solidified alloy had an irregular shape, but it was spheroidized in the rapidly solidified material, Figures 4 and 5. The dispersed phase particle diameter in the rapidly solidified alloy was on the order of 0.2 to 1.5 microns. Cusps occurred on the surfaces of the particles at intersections with grain boundaries and the grain boundaries appeared to be pinned by the particles.

The grain size of the rapidly solidified and extruded ternary alloy, Figure 6, was finer than that of the binary alloy in the corresponding condition. The dispersed phase was irregular in shape, but a tendency for spheroidization was evident. Cusps were observed at the intersection of particles and grain boundaries.

The cold working and annealing treatment reduced the grain size of the rapidly solidified binary alloy markedly to approximately 2.5 to 5 microns, as shown in Figure 7. After this same treatment, the ternary alloy had a grain size of approximately 1.5 to 4 microns, as shown in Figure 8. The carbon steel had a ferrite grain size at this stage of 1.5 to 3 microns, as shown in Figure 9. However, the microstructure was not optimum for this alloy since some massive carbides present in the extruded material persisted, and the volume fraction of very fine carbides would consequently be reduced from the optimum amount, as shown in Figure 10.

Figures 11 to 13 shown that the grains of all three alloys grew during the slow strain rate test. The grain size of the control specimen grew by a factor of roughly 3, which is consistent with reported results by Oleg D. Sherby, even through the microstructure was not optimum prior to the test. The grains of the ternary alloy grew by a smaller factor so that the final grain size of the ternary alloy was finer than that of the control specimen. The grain size of the binary alloy was approximately equal to that of the control.

In the next report period, the processing and evaluation of the alloys already started will be continued and new alloys melted as indicated by the results. While the experimental techniques are now capable of ranking substantially different alloys in order of merit, it would be desirable to improve the accuracy and precision of the technique in order to discriminate between alloys with smaller differences. Several small changes in the experimental techniques are contemplated.

* The term "dispersed phase" will be used until such time as the phase in question has been identified. The equilibrium boron rich phase in the binary alloy is Fe_2B, but a nonequilibrium phase may have formed as a result of the rapid solidification. If a nonequilibrium phase were to form during rapid solidification, it might undergo a transformation or a series of transformations during subsequent processing. Also, as the program progresses and alloying elements are added, even the equilibrium phases will generally be unknown.

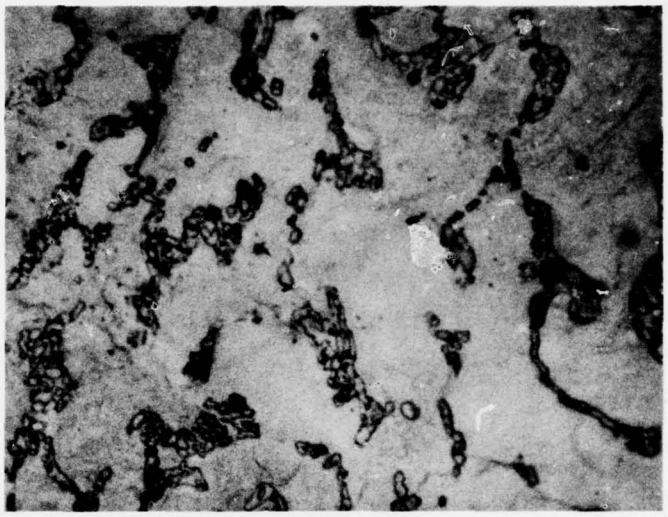


Figure 4. Slowly Solidified and Extruded Binary Alloy

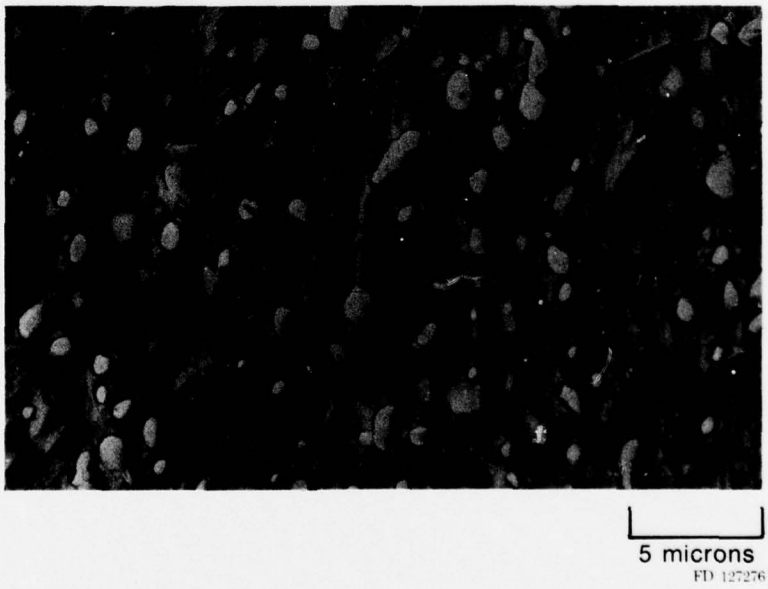


Figure 5. Rapidly Solidified and Extruded Binary Alloy

Figure 6. Rapidly Solidified and Extruded Ternary Alloy

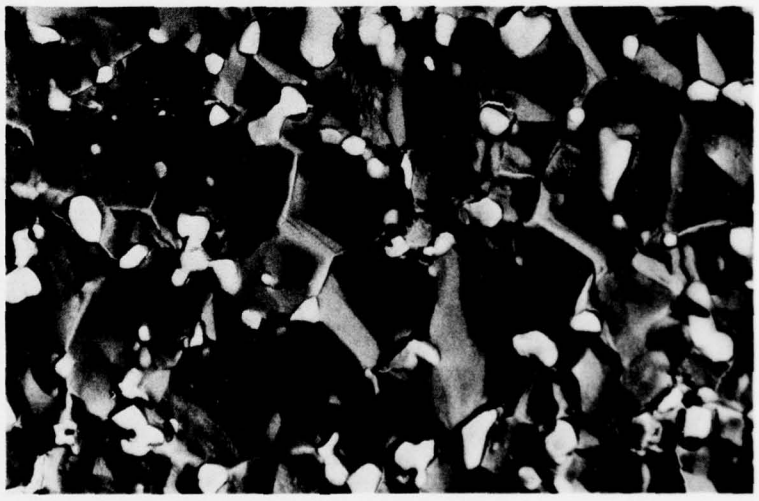


Figure 7. Recrystallized Binary Alloy

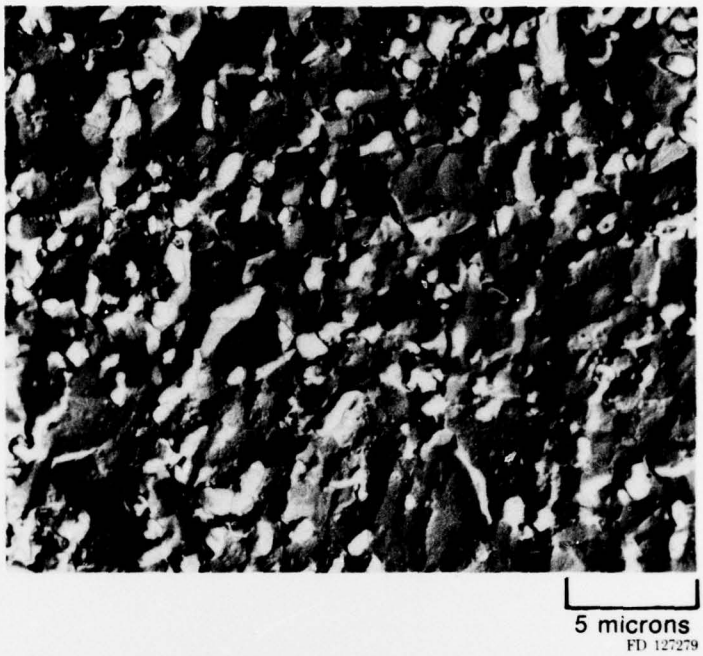


Figure 8. Recrystallized Ternary Alloy

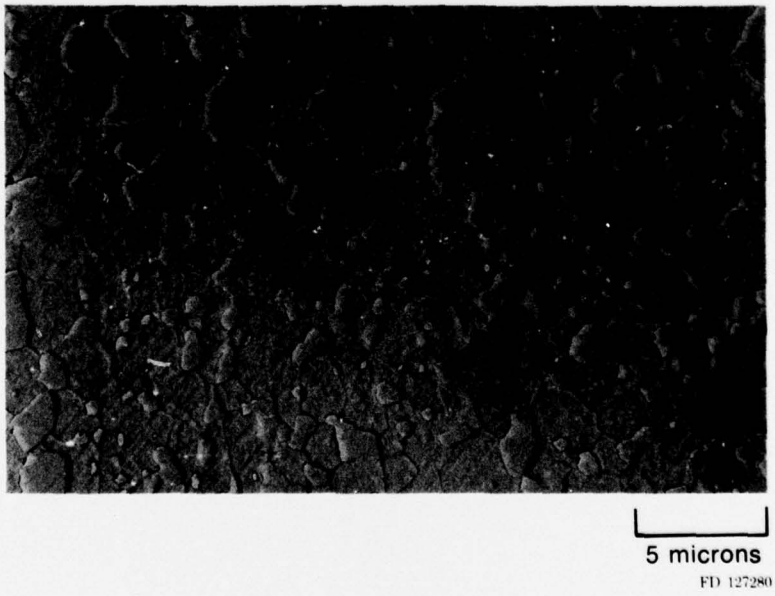


Figure 9. Recrystallized Ultrahigh-Carbon Steel

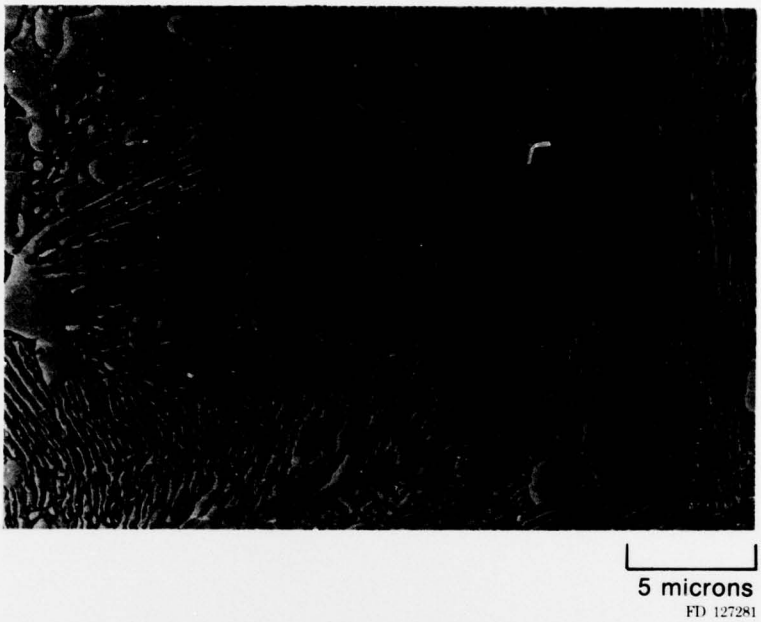


Figure 10. Extruded Ultrahigh-Carbon Steel

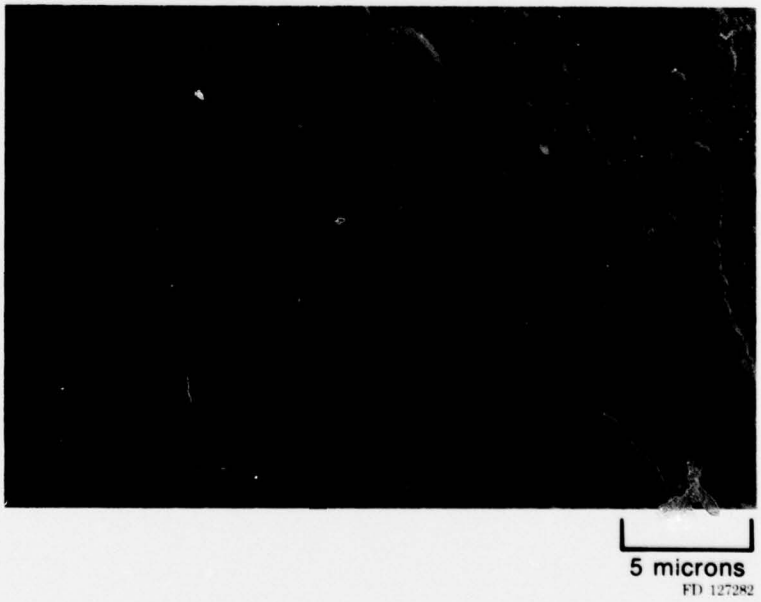


Figure 11. Ultrahigh-Carbon Steel After Slow Strain Rate Test

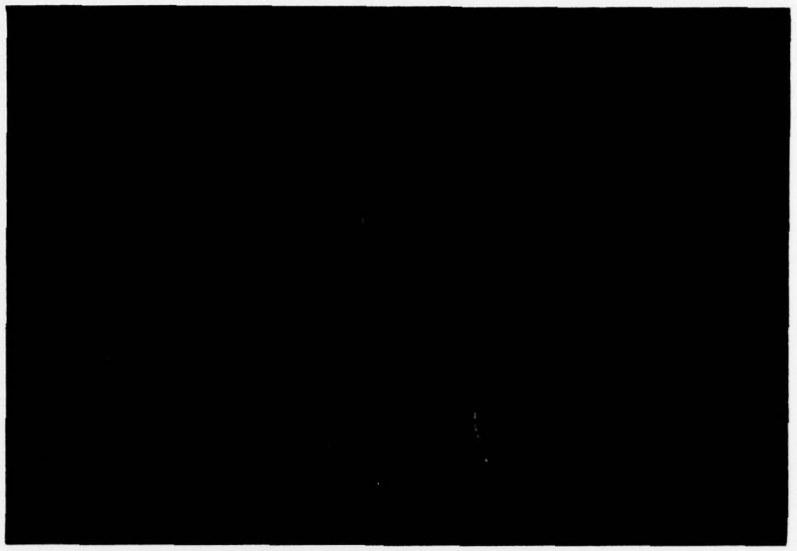


Figure 12. Binary Alloy After Slow Strain Rate Test

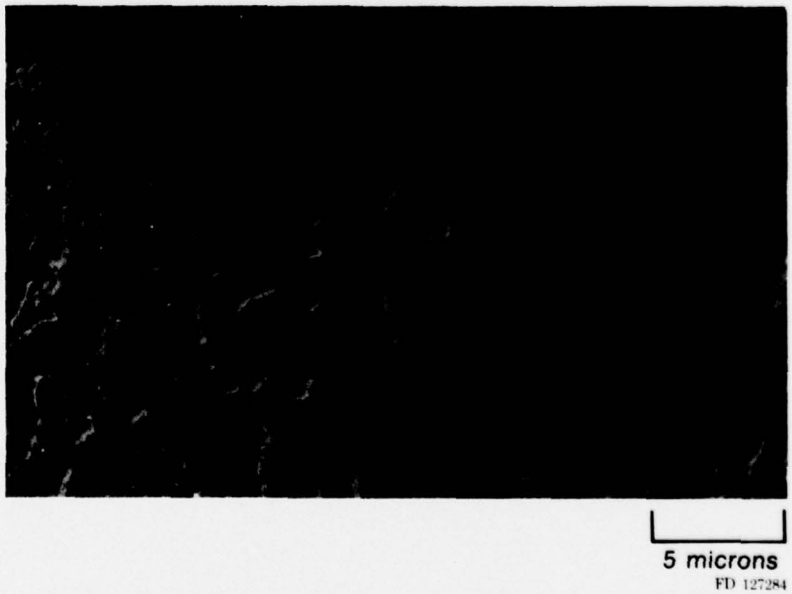


Figure 13. Ternary Alloy After Slow Strain Rate Test

SECTION III

EXPERIMENTAL TECHNIQUE

A method for producing rapidly solidified layers on the surface of alloys using a continuous laser is called laser glazing and has been described elsewhere.[2]

Laser glazing has two outstanding merits for this alloy development program. It is convenient; lots of several alloys can be treated quickly and it is capable, in principle, of producing a wide range of predetermined solidification rates, including those that encompass the highest solidification rates likely to be attained for large quantities of alloys. However, the method has limitations as well.

Some of the difficulties with laser glazing have been caused by: (1) variations in adsorbed power; (2) the brief interval the material is molten; and (3) the small cross section of the treated alloy.

For convenience, the alloys have been laser treated in lots, ordinarily 36 alloys per lot. To date, two lots have been treated for this program. During the treatment of the first lot, the laser apparently behaved erratically. The melted layer varied in depth from 0.003 inch to 0.1 inch for one set of laser glazing parameters and from 0.001 inch to 0.012 inch for the other. The depths of the second lot indicated that the erratic behavior had been eliminated. Thus, the erratic behavior of the laser presented a problem only in interpreting the results of the first lot, which included the three alloys, iron with 1% boron, iron with 0.7% molybdenum and 1.0% boron, and a 1.6% carbon steel. Since the soldification rate varies inversely with the square of the depth, variations in the depth for the first lot corresponded to large changes in solidification rate.

An analysis of the depths of the laser treated alloys of the second lot indicated statistically significant component of variation of depth with both boron content and base alloy composition as shown in Table 1. This variation is thought to be due to variations in the adsorptivity with composition and to variations in their thermal properties, especially their melting point, conductivity and specific heat. Obviously, this type of variation could be reduced greatly by measuring the depth for each alloy for a given treatment and repeating the laser treatment with suitably modified parameters. However, that approach would introduce a considerable delay. An alternative approach to the problem has been adopted for the next lot of alloys; the lot will be treated with two sets of laser glazing parameters, i.e., two scanning speeds at a constant power density, intended to yield different solidification rates. It is expected that, although the solidification rate for a given speed will vary between alloys, different scanning speeds will produce in each alloy solidification rates that will encompass those obtained in powder manufacturing processes.

Inspection of the data of the second lot indicated that the variance of depth was roughly proportional to the mean. For the depths of interest, the variation in depth would cause the solidification rate to vary by a factor of approximately two.

The scanning rate of the laser beam is on the order of 5 ft/min to 50 ft/min and the resulting dwell times are a fraction of a millisecond. In this brief interval, refractory phases may not dissolve or melt. Also, even if the pool is homogeneous, the laser treated material may not be of the bulk alloy composition because the scale of the segregation in the button ingot may approach the volume of the molten pool.

[2] Breinan, E. M., B. H. Kear and M. Banas, "Processing Materials With Lasers," Physics Today, November 1976

To avoid these difficulties the following procedure was adopted. A first laser pass was made at a low scanning speed; this deepened the pool to create a larger, more representative sample and allowed more time for the refractory phases to melt or dissolve. The second pass, which was superimposed on the first, was shallow to produce the desired solidification rate. The superimposed passes produced easily distinguished microstructures before mechanical working, but the identity of the passes was lost after much working. Other possible solutions to the problem are also being studied.

TABLE 1. DEPTH OF LASER PASS, TEN THOUSANDTHS INCH

	Boron, weight %					
Base Alloy	*0.1*	*0.18*	*0.32*	*0.12*	*1.0*	*Row Mean*
Fe	30[1]	61	51	46	65	
	56[1]	51	73	48	53	54.7
	29[1]	74	75	50	58	
Fe	47	62	72	61	75	
+	45	65	83	[2]	70	64.3
0.4% C	47	71	69	62	71	
Fe	83	58	55	59	45	
+	60	50	45	58	46	56.6
1.0% Mn	65	61	49	62	53	
Fe	74	56	50	55	64	
+	67	60	61	54	[2]	61.1
0.4% C	64	60	71	56	64	
1.0% Mm						
AISI	43	157	143	131	186	
4140	51	154	153	116	177	129.7
	48	138	176	102	171	
Fe	56	61	63	94	69	
+	46	61	71	80	69	66.8
1.5% Si	46	66	59	87	74	
+						
2.5% Al						
Column Mean	53.2	75.9	78.8	71.8	82.9	

[1] Random measurements on three laser passes on each button ingot.

[2] Invalid data with an assignable cause — insufficient shielding gas during Laser glazing.